Afdeling Algemene Chemie 1986-07-10

RAPPORT 86.72 Pr.nr. 505.6050

Onderwerp: Verslag van het "International Symposium on Near Infrared Reflectance Spectroscopy" Scheveningen, 16-17 april 1986

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8672

Afdeling Algemene Chemie 1986-07-10

RAPPORT 86.72 Pr.nr. 505.6050

Projekt: Ontwikkeling methoden van onderzoek voor voedings- en voedermiddelen met behulp van NIRS.

Onderwerp: Verslag van het "International Symposium on Near Infrared Reflectance Spectroscopy". Scheveningen, 16-17 april 1986.

Bijlagen: 1. Programma NIRS-symposium
2. RIKILT-voordracht
3. Samenvatting van alle lezingen.

Verantwoordelijk: drs N.G. van der Veen
Samenstellers : R. Frankhuizen en drs N.G. van der Veen

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Inleiding

Technicon Instruments Corporation organiseert vanaf de introduktie van de nabij-infrarood-reflectie spectroscopie (NIRS) apparatuur jaarlijks een internationaal symposium.
Het ene jaar vindt dit symposium plaats in het Technicon Science Center, het hoofdgebouw van Technicon International te Tarrytown, New York, USA en het andere jaar in een Europees land. Dit jaar werd het symposium in Nederland gehouden.
Aan dit (negende) Internationale NIRS symposium werd door ca 400 personen, waarvan ruim de helft afkomstig uit Nederland, deelgenomen.
31 deelnemers, afkomstig uit 11 landen, werkzaam zowel in het bedrijfsleven als bij overheidsinstituten en universiteiten verzorgden een lezing. Deze lezingen werden gehouden in het Kurhaus te Scheveningen. Op woensdag 16 april werd een plenaire sessie gehouden en op donderdag 17 april een drietal parallelsessies te weten: een Agriculture, een Food processing en een Pharmaceutical/Chemical sessie (zie voor een volledig programmaoverzicht bijlage 1).
Uitnodigingen van Technicon Frankrijk (in Parijs ligt de hoofdvestiging van Europa en deze organiseert alle Europese symposia) werden geaccepteerd voor het verzorgen van een lezing door dhr. R. Frankhuizen in de Food Processing Sessie omtrent de bepaling van de samenstelling van gehakt m.b.v. NIRS en het voorzitterschap van één van de agriculture sessies door drs N.G. van der Veen.
Voor de volledige tekst van de RIKILT lezing wordt verwezen naar bijlage 2.
Omdat Technicon bij aanvang van het symposium - in tegenstelling tot andere jaren - een bijna compleet overzicht klaar had van de samenvattingen van alle lezingen (zie bijlage 3) zullen hier alleen enkele saillante onderzoekresultaten en meningen vermeld worden.

Prof. dr K. Molt, University of Duisburg, Germany, heeft FT-IR vergeleken met NIRS. Hiervoor maakte hij gebruik van een groot aantal samengestelde pharmaceutische mengsels.
Van de specifieke pieken in het midden-IR-gebied bleef in het NIR-gebied weinig over, hoewel een aantal boventonen en combinatietonen van fundamentele vibraties goed herkenbaar waren in het NIR-gebied. De berekeningen met NIR-data van de samenstelling van de

mengsels had hem in sterke mate verrast.
Gemiddeld waren de resultaten behaald met NIRS een factor 10 beter dan die met FT-IR. Dit komt niet door verschillen in monstervoorbereiding. Deze resultaten werden door Dr. D.E. Honigs (University of Washington, Seattle - USA) en Dr. T. Hirschfeld (Lawrence Livermore National Laboratories, University of California - USA) bevestigd. Laatstgenoemde is de mening toegedaan dat met FT-NIR gelijke en wellicht betere resultaten zijn te behalen dan met NIRS voor wat betreft kwantitatieve analyse, maar dat door het ontbreken van de juiste meetoptiek en software FT-NIR in de praktijk nauwelijks wordt toegepast.
FT-IR is voor kwantitatieve analyse van samengestelde voedingsmiddelen nauwelijks bruikbaar vanwege het ontbreken van het lineaire verband tussen absorptie en concentratie en de zeer hoge energie waarmee het monster aangestraald wordt. Ook via polynome regressie-analyse valt niets beters te verwachten omdat deze rekentechniek veel te veel tijd nodig heeft. Prof. Molt liet verder zien dat NIR-berekeningen op basis van log $^{1}/_{R}$-waarden betere resultaten gaven dan berekeningen gebaseerd op Kubelka-Munk grootheden.
Dr Honigs merkte op dat het fundamentele verschil tusen NIR en IR in de mathematiek ligt en dat de meeste mathematische rekentechnieken voor NIR-data nagenoeg dezelfde resultaten opleveren. Wel vindt hij "Principle Component Analysis" (PCA) sneller en robuuster dan "Multiple Linear Regression Analysis". Bij PCA wordt van de verkregen spectra eerst het gemiddelde spectrum afgetrokken waarna met de residuelen (spreidingen) verder wordt gerekend.
Dit betekent dat er (n-1)assen (onafhankelijk variabelen) nodig zijn om de populatie (monsters) volledig te beschrijven. In de praktijk blijkt dat veelal volstaan kan worden met veel minder assen om grootheden te voorspellen.

Dr Hirschfeld belichtte in zijn lezing het brede toepassingsgebied van NIRS. Naast de bepaling van de samenstelling van produkten kunnen ook niet-chemische eigenschappen bepaald worden, zoals de dichtheid, viscositeit, elasticiteit, baktemperatuur etc. Het blijkt dat "no obvious spectral peaks are required". De methode is niet geschikt voor "trace analysis".

Transmissiemetingen

In een persoonlijk gesprek is de mening en ervaring gevraagd aan Honigs en Hirschfeld betreffende transmissiemetingen. Hoewel beiden wel enige toepassingsmogelijkheden voor transmissiemetingen zien voorspelden zij voor NIRS-transmissiemetingen geen grote toekomst, omdat de lichtintensiteit dermate klein is dat van het doorstralen van objecten, als b.v. tomaten, amper sprake is. Hirschfeld meent dat het licht, gemeten op de detectors, grotendeels afkomstig is van diffuus licht dat rond het object loopt i.p.v. er doorheen. In die gevallen waar resultaat geboekt is (Kaffka, Centre of Food Control, Budapest) gaat het om het kortgolvige gebied (600-800 nm). In dit gebied is de lichtintensiteit groter, maar van enige specifieke absorptie is amper sprake. Het gaat meer om een algemeen verschil in absorptie niveau. Door bij verschillende golflengten te meten kunnen dan correlaties berekend worden tussen absorptie en concentratie van enige componenten. Hirschfeld waarschuwde voor een te grote verwachting van de praktische mogelijkheden van NIRS-transmissie metingen.
Dhr. Y. Mulard (Product manager Technicon International) verklaarde desgewenst dat er bij Technicon niet gewerkt wordt aan transmissie metingen. Hoewel zij enig onderzoek in die richting hebben gedaan met redelijk resultaat (doorstralen van halve appels) zien zij met name problemen in de praktische uitvoering. Gebruikers staan een analyse snelheid voor ogen (tientallen metingen per min.) waaraan Technicon onmogelijk kan voldoen. Hij waarschuwde voor optimistische geluiden van andere fabrikanten (Neotec) die een totale scan in zeer korte tijd kunnen opnemen (0.2 sec.). Om tot een redelijke betrouwbare analyse te komen moeten met deze apparatuur vele tientallen, zo niet honderden scans uitgevoerd worden, hetgeen bij met name on-line metingen ongewenst is.
Een ontwikkeling die volgens Hirschfeld gaande is en ook door Technicon nagestreefd wordt is verdere optimalisering van de mathematische software programma's en een ontwikkeling op het gebied van fiber optics. Laatstgenoemde ontwikkeling geeft de mogelijkheid de meting zelf uit te voeren buiten de apparatuur (Honigs heeft al over een afstand van 100 m metingen uitgevoerd). Een van de voordelen van deze techniek is dat de meting direct aan een object uitgevoerd kan

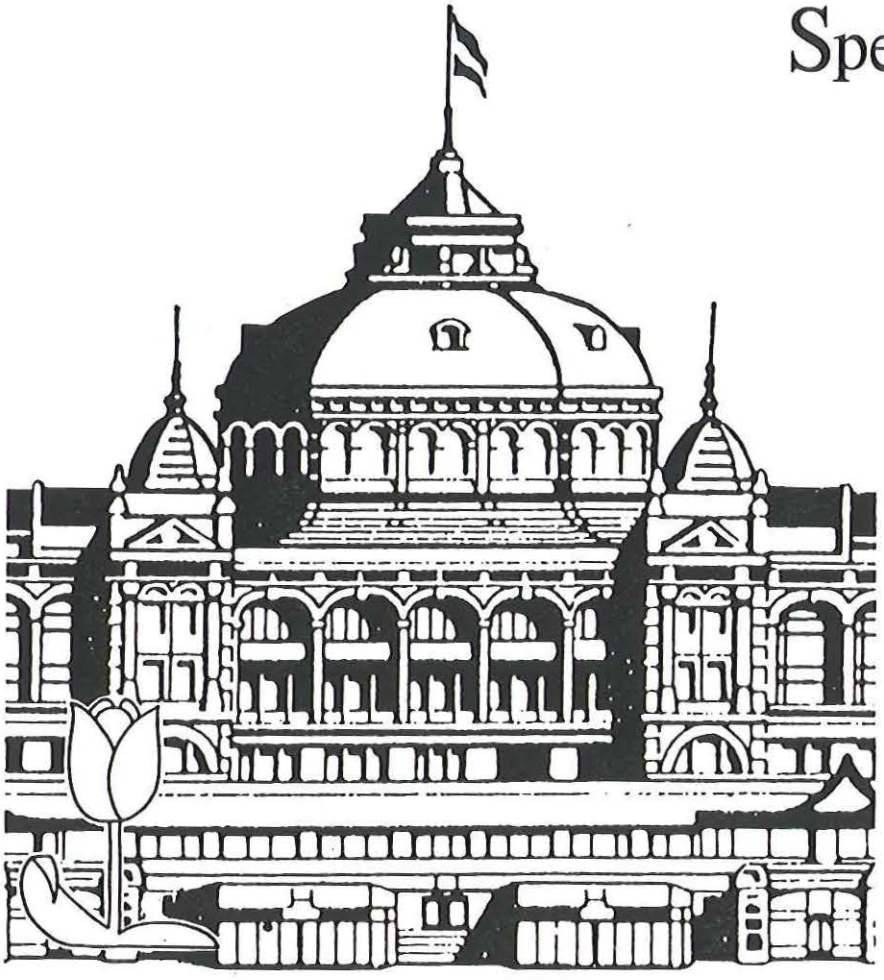
worden, hetgeen bij grote monsterobjecten (b.v. meloenen, hammen etc.) grote voordelen biedt.

Samenvatting

Samenvattend kan gesteld worden, dat het symposium veel waardevolle informatie heeft opgeleverd. De organisatie was perfect en het niveau van sprekers over het algemeen zeer hoog. Met name de informele contacten met NIRS-specialisten als dr Honigs en dr Hirschfeld hebben praktische informatie opgeleverd, welke de richting van enkele onderzoekprojekten op het RIKILT zullen beïnvloeden en tot nog betere resultaten zullen leiden.

Technicon International Symposium

Near Infrared Reflectance Spectroscopy



Scheveningen 16-17, April 1986

Program

GENERAL INFORMATION

REGISTRATION:

The registration desk will be open in the "Foyer"

- Wednesday 16th. from 09.30
- Thursday 17th. from 08.30

Please do not register at the last minute.

You will receive the final program, your badge (necessary to pass lunch control).

INFORMATION DESK:

The information desk will be open

- on Wednesday from 09.30 till 17:30
- on Thursday from 08.30 till 16:30.

MEALS

Wednesday and Thursday, lunch will be served in "Kurzaal" restaurant.

Wednesday evening, Technicon offers you a gala dinner in the salons 1 - 2 of the Kurhaus.

SIMULTANEOUS TRANSLATION:

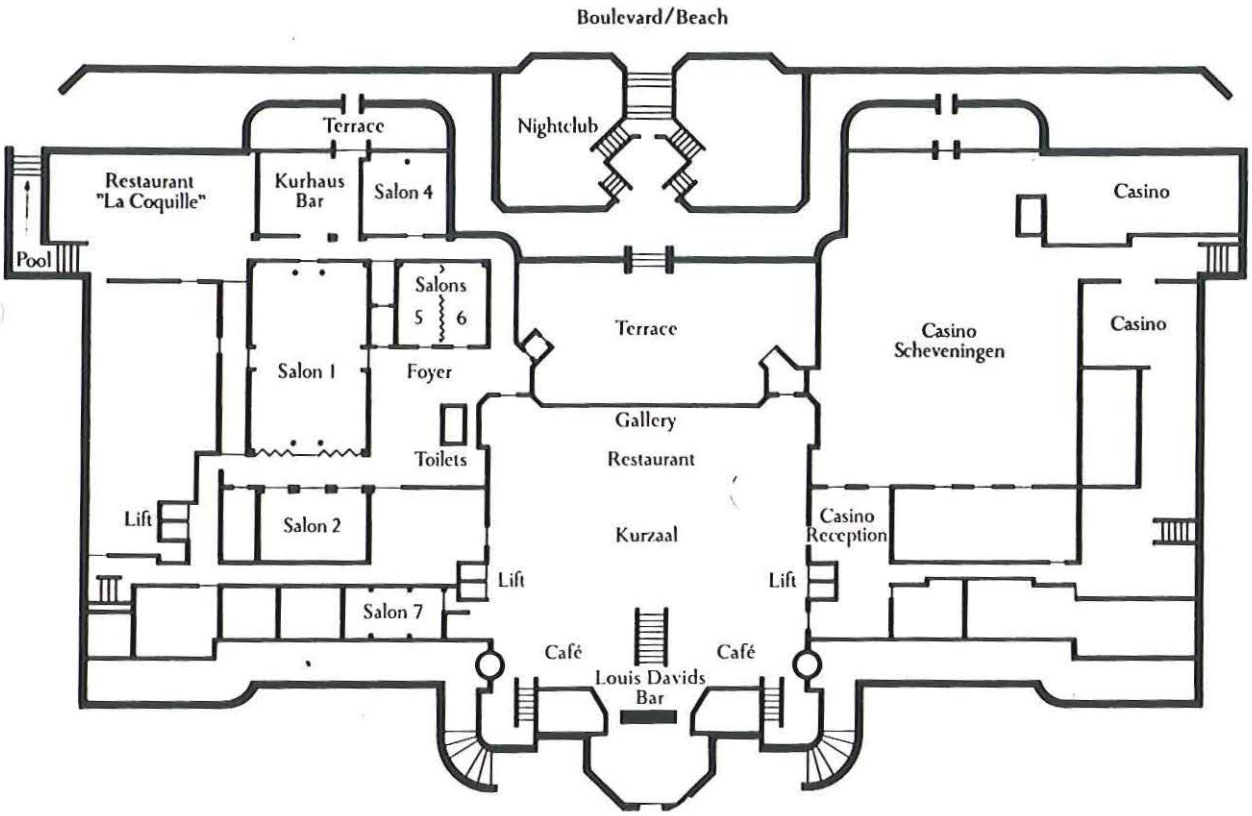
Plenary session : translation from English to German and French

Agriculture session : translation

- from English into German and French
- from French into English and German.

TELEPHONE:

Public telephone are available at the hotel reception desk.



Salon 105
Salon 107
Gallery

Follow the Signs

Salon 1	– Plenary session – Agriculture session
Salon 2	– Food processing session
Salon 5+6	– Pharmaceutical/Chemical session
Salon 7	– Infranet and new systems *(Workshop I)*
Salon 105	– Sample selection/sample handling *(Workshop II)*
Salon 107	– New software. *(Workshop III)*
Foyer	– Registration, Information
Gallery	– Exhibition

Wednesday 16th April		
Morning	Opening Plenary session Salon 1	
Afternoon	Plenary session Salon 1	Workshops I. Salon 7 II Salon 105 III Salon 107

Thursday 17th April		
Morning	Pharmaceutical chemical session Salon 5 + 6 Food processing session - Salon 2 Agriculture session - Salon 1	Workshops I. Salon 7 II Salon 105 III Salon 107
Afternoon	Plenary session Salon 1	Workshops I. Salon 7 II Salon 105 III Salon 107

Wednesday April 16, 1986

9.30 Registration

11.00 Opening Address

M.S. Day
Vice-President Marketing Industrial Systems, Technicon International Division

PLENARY SESSION

Chairman: Prof. A.J.H. van Es
I.V.V.O., Wageningen - The Netherlands

The role of NIRS alongside other analytical techniques
Dr. T. Hirschfeld
Lawrence Livermore National Laboratories, University of California - USA

The evolution and development of NIRS in Europe
Y. Mulard
Technicon International Division

INFRANET: a new dimension in NIRS
H.W. Volin
Technicon, Tarrytown - U.S.A.

12.45 Lunch

Chairman: Prof. G. Dijkstra
R.I.V.M., Bilthoven - The Netherlands

14.15 Common themes in NIRS mathematics
Dr. D.E. Honigs
University of Washington, Seattle - USA

Application of multidimensional analysis in NIRS
D. Bertrand
INRA Nantes - France

15.30 Break

Chairman: Dr. P.S.C. van der Plas
University of Technology, Delft - The Netherlands

16.00 Use of PIDA for agricultural products
Prof. A.J.H. van Es
I.V.V.O. Wageningen - The Netherlands

NIRS - Theoretical considerations and practical experience
Prof. K. Molt
University of Duisburg - Germany

Thursday April 17, 1986

PHARMACEUTICAL/CHEMICAL SESSION

Chairman: Dr. J.P. de Kleijn
Organon - The Netherlands

9.00 NIRS - A new impulse for the pharmaceutical analysis
Dr. G. Dertinger
Sandoz, - Germany

Routine experience with product identification
N. Augris,
Roger Bellon, (Rhône-Poulenc) - France

Real Time quality assessment using NIRS
K. Leiper
Glaxo, UK

The use of NIRS for the determination of hydroxyl value of alkoxylates
Dr. W.C. Campbell
I.C.I. Petrochemicals and Plastics Division - UK

10.30 Break

Chairman: Prof. W.E. van der Linden
University of technology, Enschede - The Netherlands

11.00 Support of antibiotic production
Dr. P. Johnson
Dista Products, Liverpool - U.K.

Control of fungicides with NIRS
D. Amiot
Rohm and Haas - France

Progress towards on-line control with feed back of organic synthesis
Dr. M. Leach
Kingston Polytechnic - U.K.

12.15 Lunch

Chairman: Dr. P.C.M. van Woerkom
Akzo - The Netherlands

13.45 NIR analysis of D and L isomers
Dr. D.E. Honigs
University of Washington, Seattle - USA

Some applications of NIRS in glasswool industry
Dr. R. Fugier
Isover - France

PLENARY SESSION

15.30 The future of NIRS
Dr. T. Hirschfeld
Lawrence Livermore National Laboratories, University of California - USA

The future of NIRS in agriculture
Dr. Ir. R. Biston
Agricultural Research Centre - Belgium

DISCUSSION ON THE FUTURE OF NIRS

16.15 Conclusions of the meeting

Thursday April 17, 1986

FOOD PROCESSING SESSION

Chairman: Dr. F.D. Tollenaar
The Netherlands

9.00 Implementation of NIR as screening aid for quality aspects of food
P.A. de Lezenne-Coulander
Food Inspection Service, The Hague - The Netherlands

Examples of use of the InfraAlyzer in the cocoa and chocolate industry
J. Pontillon
Barry - France

Use of NIRS for the quality assurance in the production of special types of food: dietetic and baby foods
Dr. F. Taccani
Plasmon (Heinz) - Italy

10.15 Break

Chairman: Dr. K. Broer
CIVO - The Netherlands

10.45 Performance of the InfraAlyzer 400D on ice cream
C. Usher
Unilever Research, Sharnbrook - UK

Use of the new InfraAlyzer 450D with a two position liquid/solid sampling drawer
Technicon France

The use of an autocontrol program in the dairy industry
W.G. Verschoor
Nestlé - The Netherlands

12.15 Lunch

Chairman: Dr. F.D. Tollenaar
The Netherlands

14.00 Some applications of NIRS in a food research institute, including packaging material analysis
A.M.C. Davies
A.F.R.C. - U.K.

In-line measurement of beer original gravity
Dr. F.H. White
Bass Brewing Ltd - U.K.

Compositional analysis of minced meat by NIRS
R. Frankhuizen
RIKILT - The Netherlands

PLENARY SESSION

15.30 The future of NIRS
Dr. T. Hirschfeld
Lawrence Livermore National Laboratories, University of California - USA

The future of NIRS in agriculture
Dr. Ir. R. Biston
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DISCUSSION ON THE FUTURE OF NIRS

16.15 Conclusions of the meeting

Thursday April 17, 1986

AGRICULTURE SESSION

Chairman: Dr. Ir. R. Biston
Agricultural Research Centre, Gembloux - Belgium

9.00 Quality determination in forage by conventional and novel mathematical techniques
G. Downey
Kinsealy Research Centre - Ireland

The Control of in-vivo digestibility of forages
Prof. A.J.H. van Es
I.V.V.O., Wageningen - The Netherlands

10.15 Break

Chairman: W.C.F. Vercauteren
CHV - The Netherlands

10.45 Quality control of feed in a large cooperative group.
C. Bernard
UCAAB - France

The use of NIRS in a large feed cooperative association
P. Petersen
L.A.G. - Denmark

Choice of specific wavelengths for glucosinolates in whole rapeseed by NIRS
V. Furstoss
INRA - France

12.30 Lunch

Chairman: Dr. N.G. van der Veen
RIKILT - The Netherlands

14.00 Development and practical use of NIRS on grains and derivates
R. Rijkers
Technical Laboratory, Rotterdam - The Netherlands

Experience of NIRS as method of payment for protein in wheat for farmers in Swedish grain trade
H. Johansson
Svalof AB - Sweden

Quality control of cereals in Spain
Dr. C. Rivas
SENPA - Spain

PLENARY SESSION

15.30 The future of NIRS
Dr. T. Hirschfeld
Lawrence Livermore National Laboratories, University of California - USA

The future of NIRS in agriculture
Dr. Ir. R. Biston
Agricultural Research Centre - Belgium

DISCUSSION ON THE FUTURE OF NIRS

16.15 Conclusions of the meeting

Workshops and group demonstrations:

Workshops 1

Demonstration of Infranet and new systems

Workshop 2

Sample selection and sample handling
- The use of PICKS program for sample selection
- Demonstration of special hardware to analyse liquids, solids, semisolids

Workshop 3

New software

- APC for calibration and prediction
- Prestat for prediction and statistics
- PIDA for product identification by discriminant analysis

Wednesday 16th				Thursday 17th		
H	W1	W2	W3	W1	W2	W3
9.00				French	German	English
10.00				German	English	French
11.00				English	French	German
14.00	French	German	English	French	German	English
15.00	German	English	French	German	English	French
16.00	English	French	German	English	French	German

EXHIBITION

Technicon INFRAALYZER 500C	:	a research instrument
Technicon INFRAALYZER 450LR	:	for the analysis of solids, semi-solids and liquids analysis
Technicon INFRAALYZER 450DR	:	for the analysis of milk and dairy products analysis Presented with the new two position liquid/solid drawer
Technicon INFRAALYZER 350	:	a simple instrument for dedicated analysis
Technicon INFRAALYZER 250	:	a simple instrument for grain trade and flour milling industries

INTERNATIONAL DIVISION
6-10, quai de Seine - 93206 Saint-Denis/France

Compositional Analysis of minced meat by NIR spectroscopy

mr.R.Frankhuizen, Dept.for Automation of Analysis, State Institute for Quality Control of Agricultural Products, Wageningen, the Netherlands

SUMMARY

Part of the activities of the State Institute for Quality Control of Agricultural Products consists of developing and promoting automated methods to measure quality parameters of foods and feeding-stuffs. In this framework the suitability of NIR to measure the chemical composition and the product specification of minced meat has been evaluated. This study shows that sample preparation plays an important role in meat analysis. Particle size, homogeneity and temperature of the samples must be standardized. Using the best combinations of three NIR-filters, useful regression curves were developed to determine moisture, fat and protein in minced meat. Determination of product species, even qualitatively, is hardly possible when using the current NIR equipment. Details and results of this feasibility study will be presented.

1 INTRODUCTION

Part of the activities of the State Institute for Quality Control of Agricultural Products consists of developing and promoting methods to measure quality defining parameters in foods and feeds. Because a lot of samples have to be analysed on composition and quality aspects, there is a need for fast methods of analysis, especially for methods for which only a minimum of sample preperation is required and a good comparability with reference methods is obtained. Near infrared reflectance spectroscopy (NIR) instruments

particularly have the potential to provide these benefits. Since 1980 an Infra Alyzer-400 is used at our institute for the compositional analysis of milk powders, cheese and feeding-stuffs on a routine basis and since 1984 an Infra Alyzer-500 is used for research purposes and for determining calibrations for filter instruments. The suitability of NIR for measuring the chemical composition and the product specification of minced meat samples was investigated last year. The Dutch Food and Drugs Act regulates the percentage of fat and the moisture/ protein ratio in minced meat. The quality of minced meat is defined by the quality of the raw materials, the beef/pork ratio, the fat content etc. The producers of meat products are faced with a great variety of raw meat materials and therefore it is difficult to keep the product quality constant. The only way to ensure constant quality is to analyse the composition of the raw materials and produced products during processing as fast as possible. On basis of these results the producer can re-adjust the production process. Fast methods of analysis such as NIR can be of great help in this respect.

2 EXPERIENCES WITH THE SAMPLE PREPERATION OF (MINCED) MEAT SAMPLES

Previous to the analysis of the chemical composition the influence of grinding, temperature and packing of the samples in the sample cup on the accuracy and reproducibility of NIR measurements is evaluated. For that purpose a great number of measurements on raw and freeze-dried beef and pork meat samples have been performed. The measurements were both carried out in an open sample cup and in a sample cup closed with a quartz window. Mathematical transformation of the raw absorbance data to first and second order derivatives was done to reduce the influences of particle size, sample temperature, sample

temperature, sample compactness and intercorrelations between wavelengths on the measurements. Repacked samples were used to measure repeatability of raw data and first and second order derivatives. In diagram 1 relevant factors are given for the sample analysis. It can be resumed that the accuracy of the NIR measurements of minced meat samples are highly influenced by the sample homogeneity, sample temperature and sample compactness. By standardization of the sample preperation and averaging the log 1/R values of three repacked samples, satisfactory results are obtained.

Diagram 1: Important factors for sample analysis.

-The sample must be representative for the meat lot. Grinding or cutting is necessary to get a homogeneous sample. At high temperature loss of moisture and spreading of the fat particles may give problems.

-Standardization of the temperature is very important in connection with shifts of the water peak in the spectrum.

-Measure three times to obtain a desirable accuracy.

-Closed cups gives smear on the window, so sample packing in a open sample cup is inevitable.

-Calculations must be carried out with raw data, because derivative spectra of meat have a poor signal to noise ratio.

Diagram 2 gives a standardized method used for the analysis of the composition of minced meat samples.

Diagram 2: Standardized method of analysis for (minced) meat samples.

-Take about 200 g of raw fresh meat. (representative for the meat lot).

-Cool to about 5°C

-Cut in peaces of about 4 cm2. Homogenize in a Robot-Coupe meat cutter for 12 sec at 1500 rpm and 18 sec at 3000 rpm.

-Leave samples to them to room temperature (21±2C)

-Measure the samples in triplicate in open cup

-Carry out calculations with the triplicate average of the raw (log 1/R) data.

Diagram 3 gives the standard deviation (Sc) calculated for the average of three measurements of raw beef in an open sample cup. Figure 3a shows the range of ten measurements of raw beef in an open sample cup.

Diagram 3: Standard deviation of the measurement.

Standarddeviation Sc of the measurement when the sample cup is packed several times:

Sc= range x 0.325

Variation for ten measurements = 45 (mil.log.abs.)

Maximum absorbance range = 1300 (mil.log.abs.)

Relative variation = 45/1300 x100%= 3.5%

Sc= 3.5 x 0.325 = 1.14%.

Sc for the avarage of three measurements = $1/\sqrt{3}$ x Sc = 0.7% relative.

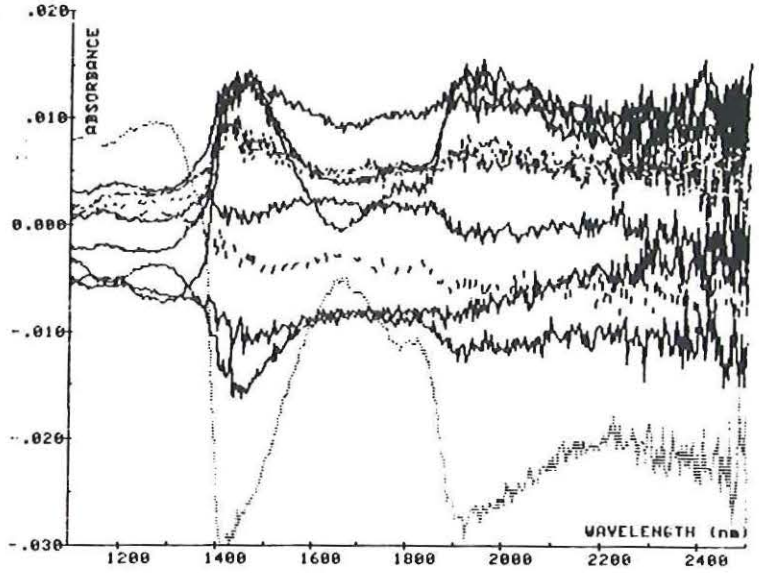

Fig.3a Variation in absorbance for measurments of raw beef ten times repacked in an open sample cup.

3. Results of compositional analysis.

45 Samples of minced meat were collected, prepared for analysis as given in diagram 2 and analysed for the main components by reference methods and measured by NIR. Reflectance measurements were obtained by using a Technicon Infra Alyzer-400. Reflectance data were analysed using a multiple linear regression program called "all combination search". Using the best combinations of three NIR-filters for each component, regression equations were obtained for the determination of moisture, fat and protein in minced meat.

For moisture a multiple correlation coefficient (R) was calculated of 0.99 with a standard error of estimate (SEE) of 0.94%. The percentage of moisture in the samples ranged from 42,0 to 68,8%. This standard deviation seems rather large. Which may be caused by the presence of a lot of connective tissue in the meat samples (porc-rind and tendons). This can have a negative influence

on the accuracy. Differences in salt concentrations and temperature give shifts of the water peak in the NIR spectrum and have also a negative influence on the accuracy. Therefore a wavelength of 1445 nm was selected instead of the most specific wavelength of 1940 nm. A linear correlation coefficient of 0.88 was found. 0.88. The wavelength of 1722 nm and 1759 nm were selected respectively as a reference wavelength and as a wavelength correcting for the influence of fat on the moisture absorbance. (see fig.1)

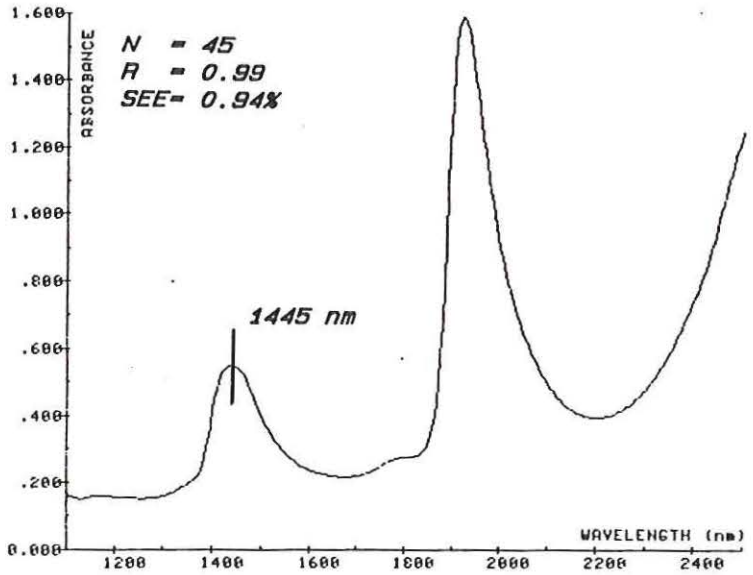

Fig.1 NIR reflectance spectrum of water showing the selected water absorption wavelength for predicting moisture in meat at 1445 nm.

As most significant wavelength (i.e. filter) to measure fat the wavelength at 1734 nm was selected Figure 2 shows the NIR spectrum of meat fat. The most specific wavelengths are 2310 nm and 2347 nm. However these wavelengths are not selected because in this region the water absorbance largely overlaps the absorbance of fat. The wavelength of 1680 nm was selected as a reference wavelength and

the 2208 nm wavelength corrects for the influence of protein on the fat absorbance. A multiple correlation coefficient of 0.99 was calculated with a SEE of 0.87. (see fig.2)

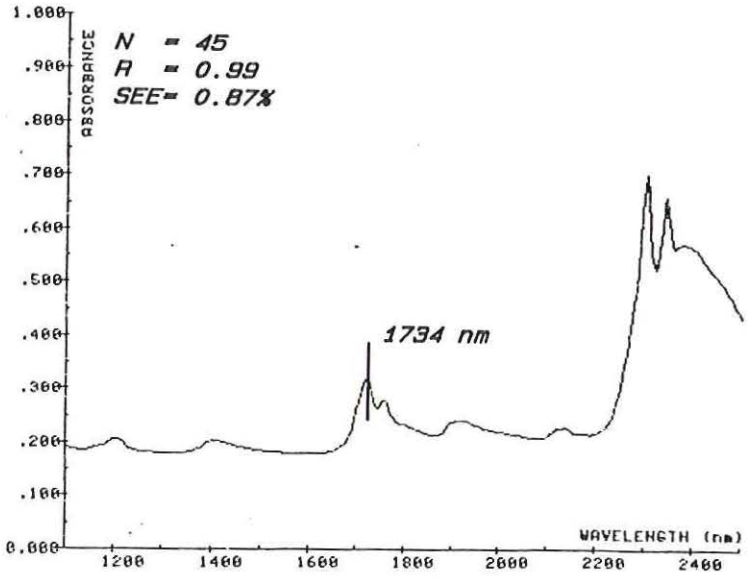

Fig.2 NIR reflectance spectrum of meat-fat showing the selected fat absorption wavelength for predicting fat in meat at 1734 nm.

For protein a multiple correlation coefficient of 0.97 was calculated with a SEE of 0.46%. The most significant wavelength for protein was 2208 nm. 2230 nm was selected as a reference wavelength and 1445 nm as a wavelength correcting for the influence of water on the protein absorbance. (see fig.3)

The calibration equations for moisture, fat and protein were tested with twenty samples of divergent composition. No significant differences were found between the standard error of estimate and the standard error of prediction.

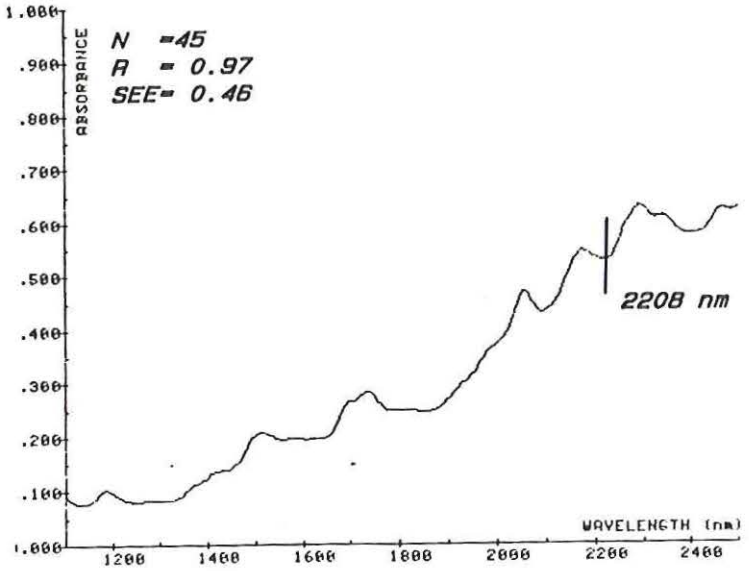

Fig.3 NIR reflectance spectrum of meat-protein showing the selected protein absorption wavelength for predicting protein in meat at 2208 nm.

4 Possibilities for meat speciation

With a number of samples of raw and freeze-dried beef and pork meat and mixtures of them the possibility of discrimination between beef and pork meat with NIR was investigated. Figure 4 shows the linear correlation as a function of the wavelength for the calibration set. High correlations are situated in the region of 2200 nm to 2500 nm. However the width of these correlation peaks are too small. A little shift in the spectra, caused by matrix influences, can already give incorrect results. Useful wavelengths seem to be situated between 1500 nm and 1700 nm and between 1100 nm and 1300 nm. The differences in spectra of raw beef and pork meat however are very small and hardly specific. (see fig.5) The first and second derivative spectra also give no better

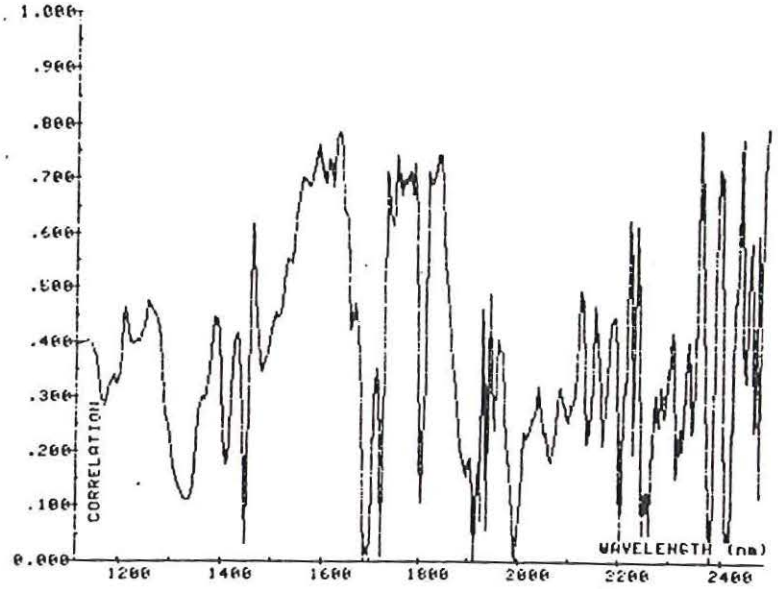

Fig.4 Correlation diagram of the calibration set for predicting the percentage of beef in minced meat.

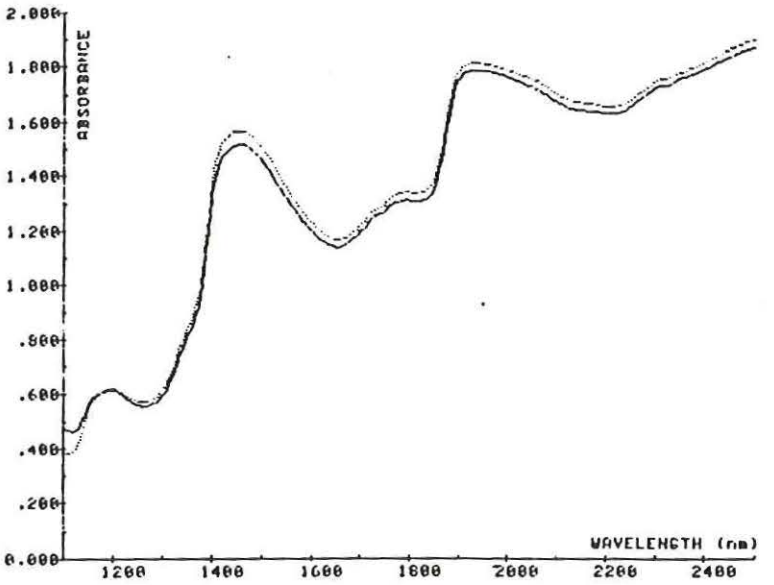

Fig.5 NIR reflectance spectra of beef (lower curve) and pork (upper curve).

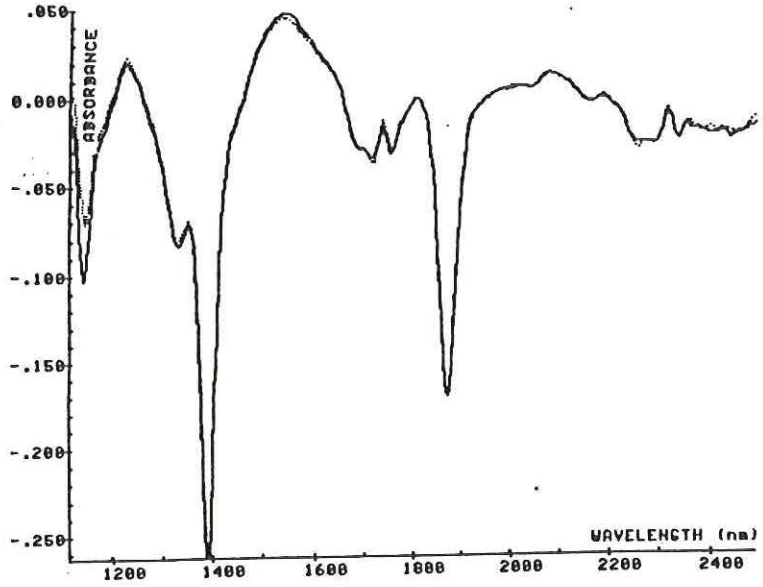

Fig.6 First derivative spectra of beef (lower curve) and pork (upper curve).

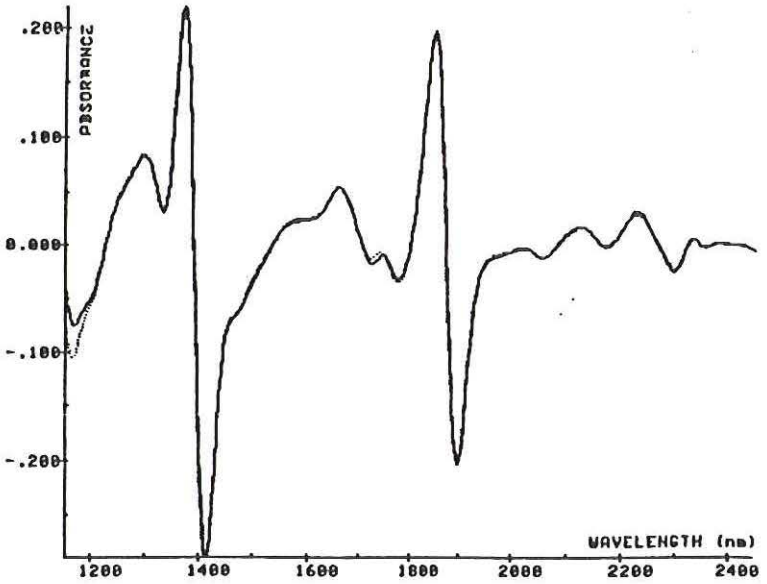

Fig.7 Second derivative spectra of beef (lower curve) and pork (upper curve).

results. (fig.6 and fig.7) Although spectra of freeze-dried beef and pork meat show as aspected, sharper peaks with a lower maximum absorbance in relation to the raw beef and pork meat spectra, again specific peaks are not found. This can be caused by loss of meat characteristics, such as structure and pigment, during freeze drying. From a number of calculated correlation curves the first derivative calibration curve with three wavelengths give the best results. The regression coefficients however are too large for reliable results owing to the fact that small absorbance differences have too be multiplied by these regression coefficients. This is confirmed with a testset of ten unknown samples of beef and pork meat. No correlations are calculated between the predicted percentages of beef and the real percentages. It can be concluded that it is not possible to differentiate between meat species by NIR analysis. Maybe more possibilities arise when the wavelength region is extended to the shorter wavelengths (up to 600 nm) and when other software such as the Product Identification Discriminant Analysis program (P.I.D.A) is used.

conclusions

-Sample preperation plays an important role in meat analysis. Particle size, homogeneity and temperature of the samples have to be standardized.

-The best results for determining the chemical composition of meat are obtained when raw NIR-data (log 1/R) are used. Derivative spectra give a signal to noise ratio which is too high.

-When the sample preparation is standardized useful regression equations can be generated to determine the percentage of moisture, fat and protein in minced meat.

-Determination of product species, even qualitatively, is not possible when using the current NIR equipment.

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ABSTRACTS

NIRS SYMPOSIUM SCHEVENINGEN 16-17 APRIL 1986

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ABSTRACTS

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NIRS International Symposium Scheveningen 16-17, April 1986

THE EVOLUTION AND DEVELOPMENT OF NIRS IN EUROPE

Y. MULARD, TECHNICON INTERNATIONAL DIVISION, FRANCE.

NIRS was introduced in Europe in 1974 for the analysis of the main components -moisture and protein- incereals.
It took 10 years to have the spectroscopists recognize the method as a powerful analytical technique.

The author will give the importance of this method in terms of yearly placements in Europe and then will review the industries using NIRS : grain trade, flour milling, feed milling, research, food processing, dairies, chemical and pharmaceutical.

All the technical improvements since 1974 will be reviewed to give the present status of the technique and the trends for future, user transferable calibration, analysis of liquids by transflectance, qualitative analysis for raw material identification, new mathematical techniques, analysis of micro components.

NIRS International Symposium Scheveningen 16-17, April 1986

I N F R A N E T : A NEW DIMENSION IN NIRS

H. VOLIN, TECHNICON TARRYTOWN, USA.

Since delivery of its first InfraAlyzer system in 1975, over 6000 have been installed on a worldwide basis. As the number of installations have grown, so have the requirements for centralized data accumulation and processing. This paper discusses Technicon's new InfraNet concept, a remote telecommunication network whereby data can be directly downloaded and uploaded from a variety of InfraAlyzer systems into a microcomputer workstation.
Log values are stored automatically and calibration equations are transferred via modem. Field trials in the grain segregation and food processing industry will be discussed.

N I R S International Symposium Scheveningen 16-17, April 1986

CURRENT N I R S MATHEMATICS

D.E. HONIGS, UNIVERSITY OF WASHINGTON, SEATTLE, USA

In the last few years there has been a large outpouring of new mathematics for Near Infrared Spectroscopy. Each of these new techniques promises to be better than any previous work. Yet, there are two interesting points which should be noted. First, it is exceedingly difficult to prove that there is any difference between the different mathematical techniques. Secondly, virtually every one of these techniques fall into the category of global or local calibrations. In this presentation, these points will be addressed in detail.

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USE OF PIDA FOR AGRICULTURAL PRODUCTS

A.J.H. VAN ES, J.H. WOLSINK AND H.J. VEDDER

I.V.V.O., THE NETHERLANDS

Product identification is needed when measuring large series of samples with NIRS. This is the more needed when the property to be measured does not have one or two fairly clear absorptions in the NIR spectrum such as for instance water and protein content. Digestibility of feeds for livestock is such in ill-defined property. After a short description of the PIDA program some examples will be presented of its use for predicting digestibility of feeds. The leading concept will be prevention of incorrect results. Furthermore the PIDA program was used to arrive at robust regression equations and some data will be presented to show the advantages of such a procedure.

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NIRS IN PHARMACEUTICAL ANALYSIS - THEORETICAL CONSIDERATIONS AND PRACTICAL EXPERIENCE

PROF. DR. K. MOLT, UNIVERSITY OF DUISBURG, GERMANY.

NIRS is vibrational spectroscopy and may be applied to all kinds of organic compounds. Using some pharmaceutical samples, we tried to acquire a feeling for the potential of NIRS compared to spectroscopy in the classical middle infrared (MIR). A large number of well defined 2- and 3-component mixtures were prepared. MIR-spectra of the solid samples were taken using potassium bromide disks and diffuse reflectance. These results are compared with a quantitative evaluation of diffuse reflectance NIR-spectra. Statistical data and relative advantages of both methods are discussed. Differences and problems regarding the techniques of sample preparation are demonstrated.

NIRS International Symposium Scheveningen 16-17, April 1986

NIRS - A NEW IMPULSE FOR THE PHARMACEUTICAL ANALYSIS

DR. G. DERTINGER, SANDOZ, GERMANY.

An essential element of drug analysis is the preparation of the samples for measurement. Simplification in that handling will reach a significant effect on reduction of workload in an Analytical Laboratory. It would be an ideal tool to perform testing without preliminary grinding and extraction of the material in question.

Regarding this aspect the principle of Near Infrared Reflectance Analysis (NIRA) is successfully used for qualitative and quantitative measurements in some branches of chemical industries.

Optimization of equipment, especially in connection with a highly potent calculation system, allows now a broad spectrum of applications.

It is obvious to transfer this technique also to analytical questions in Pharmaceutical Industry.

For example, NIRA can be used in that field for

- containerwise identification of active ingredients and components,
- quantitative measurements of semifinished and finished dosage forms,
- characterisation of raw- and packaging materials and
- determination of surfaces and particle sizes.

By means of some practical examples first experiences with the new analytical method are demonstrated.

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ROUTINE EXPERIENCE WITH PRODUCT IDENTIFICATION

N. AUGRIS, ROGER-BELLON (RHÔNE-POULENC), FRANCE

The InfraAlyzer 400 connected to an HP 86 computer and the PIDA software allows the routine identification of raw materials.

We have tested this method with amino-acids.

The identification is fast and reliable if the calibration is carefully and precisely developed with enough calibrant samples. Indeed, these products obtained by fermentation show variation in particle size or form of crystallisation.

When these variations are not too large, the "Ciba-Geigy" discriminant software offers the advantages of

- development of the calibration with only one sample,
- giving a particle size index (texture index) with the identification of the product being analyzed.

NIRS International Symposium Scheveningen 16-17, April 1986

REAL TIME QUALITY ASSESSMENT USING NEAR INFRA RED REFLECTANCE ANALYSIS

by
Mr K J Leiper
Central Analytical Services Manager
Glaxo Operations UK Limited, Barnard Castle

Like every other analytical technique Near Infra Red Reflectance Analysis has a viable application area but within this area it has three complementary features which make it quite unique in chemical analysis namely:

1. No sample preparation requirement.

2. Reliable optical system capable of operating in an aggressive environment.

3. A computer based data acquisition system which allows skill transfer between more and less expert users.

These characteristics allow methodologies to be developed in research units and transferred to control laboratories but more significantly, with minor software development, to make the systems more user friendly and increase the level of control, it is possible to site these instruments in production areas for operation by production staff. Thus, current Near Infra Red Reflectance Analysis instrumentation is bringing a new perspective to in-process control analysis of batch processes, by increasing the scope for obtaining incisive analytical information within the process time envelope.

This paper will describe the background to and current developments in Near Infra Red Reflectance Analysis being undertaken in our laboratories which are directed at replacing current raw material identity testing at increased frequency on a real time basis in a non-laboratory area.

NIRS International Symposium Scheveningen 16-17, April 1986

I. C. I.
PETROCHEMICALS AND PLASTICS DIVISION
RESEARCH AND TECHNOLOGY DEPARTMENT
WILTON CENTRE

USE OF NEAR INFRA-RED SPECTROSCOPY FOR THE DETERMINATION OF HYDROXYL VALUE IN ALKOXYLATES

SUMMARY

The determination of Hydroxyl Value (Number) of petrochemical products is a manpower intensive and time consuming exercise. The production of alkoxylates (ethylene oxide and/or propylene oxide polymers) is often controlled by determination of parameters related to Hydroxyl Value such as cloud point or viscosity. However the direct measurement of Hydroxyl Value is still performed on finished products in order to ensure conformity to specification. A simple but rapid procedure for Hydroxyl Value measurement has considerable potential in economic terms.

Alkoxylates of various types (alcohol and nonyl phenol ethoxylates, polyethylene glycol, polypropylene glycol and copolymers on various base materials) were obtained and characterised. A Technicon Infra-Alyzer 500 was used to establish narrow and wide range calibrations both within specific grouping of alkoxylates and across a broad range of materials. The quality of the calibrations is such as to enable classical Hydroxyl Value measurements to be replaced by NIRS determination.

The water content of these materials is also of interest. However, the water content is subject to fluctuation with time and the construction of calibrations is possible only if water contents are measured just prior to the accumulation of the spectral data on the Infra-Alyzer 500.

W C CAMPBELL
Analytical and Polymer Science Group

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N,I,R,A, IN SUPPORT OF ANTIBIOTIC PRODUCTION

DR, P, JOHNSON, DISTA PRODUCTS, U,K,

Dista Products Limited is an affiliate of the Eli Lilly Corporation involved in antibiotic, pharmaceutical and agro-chemical production. Analytical support work for the various processes involved in these production areas can essentially be described under three headings.

1. Raw material evaluation.
2. In-process control.
3. Product specification testing.

The Technicon InfraAlyzer was initially bought as an aid to productivity during raw material evaluation and the profound effects that the instrument can have in this area will be described.

Since the purchase of the apparatus opportunities for its use in a diversity of process control functions have arisen and will be described. Examples include monitoring the drying of an agro-chemical product and determining levels of active ingredient during the recovery of an animal health antibiotic. The success of these operations will hopefully encourage the development of other applications for N.I.R.A. Future projects that are envisaged such as raw material identification and the monitoring of distillation and fermentation processes will be discussed.

NIRS International Symposium Scheveningen 16-17, April 1986

PRACTICAL EXPERIENCES WITH AN INFRAALYZER 400 IN DETERMINING THE ACTIVE INGREDIENT OF A FUNGICIDE.

by D. AMIOT and Mrs. B. FORLEN
ROHM AND HAAS FRANCE - LAUTERBOURG PLANT

ABSTRACT

With a view to increasing the production and to improving delivery delays of our quality control operations, we have investigated the performance of NIR Technique for quality control of fungicides. This paper reports the use of INFRAALYZER 400 devoted to measuring Active Ingredient of Dithiocarbamate Fungicides. Both wettable powder and flowable formulations were successfully analyzed in terms of Active Ingredient.

The following paper describes in more details our practical experiences with Thixotropic Flowable Formulations. We were able to reach the high accuracy and reliability level needed by using an adapted sample cell (cell UK 1 - Ref DMT 1525) and a rigorous handling procedure.

Nevertheless, on a routine basis, the Infraalyzer technique was found too sensitive to slight process drifts or Trouble shootings and also too sensitive to formulation changes. For example, addition of a new formulation agent at a 0.1 % level has required a new calibration of the equipment with some new filters. In many cases, substancial discrepancies between Infraalyzer and the reference chemical digestion method were attributed to Process Trouble shooting.

At that time, the high sensitivity of the Infraalyzer to slight process Trouble shooting makes it difficult to consider the equipment as a reliable quality control instrument. Some investigations are carried out to better control the process and to overcome these problems.

As a conclusion, it has been shown that NIR is an attractive method for the determination of Fungicide Active Ingredient. Using the Infraalyzer 400, measurements can be carried out easily and rapidly. The results are accurate and reproducible. The measurements involve no special sampling ability or steps which may introduce errors in the wet chemistry reference method.

Nevertheless, the high sensitivity of the Infraalyzer 400 within process Fluctuations or slight formulation changes turns out to limit its use as a reliable quality control instrument in replacement of the length chemical digestion method.

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PROGRESS TOWARDS THE ON-LINE CONTROL OF ORGANIC SYNTHESIS USING NIRA

M. LEACH, KINGSTON POLYTECHNIC, U.K.

NIRA research at Kingston Polytechnic is involved with exploring the utility of I/A 400's as tools for examining the progress of chemical reactions with real time multi-component analyses. A number of systems have been examined :

1. A simple titration, involving ethanolamine and hydrochloric acid, has been followed, with NIRA giving molar concentrations of the free amine and the amine hydrochloride during the course of the reaction. A rational has been developed to eliminate cross correlation of constituents.

2. A 50L solvent recovery still (acetone/water system) with a peristalsic pump allowing the InfraAlyzer 400 to work in an on-line mode has been developed. The InfraAlyzer 400 down-loads log values to a BBC micro computer, which acts as a process controler, initiating analyses, calculating molar concentrations, timing the pump action, and setting the reflux ratio of the still.

3. The self contained InfraAlyzer 400/BBC/pump is being used to follow the course of preparative organic reactions. Work is proceeding on the esterification of acetic acid and 1-butanol (with azeotropic removal of water).

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N I R ANALYSIS OF D AND L ISOMERS

D. E. HONIGS, UNIVERSITY OF WASHINGTON, SEATTLE, USA

In fine pharmaceuticals, the purity of D and L isomers of a compound frequently have to be determined. This particular determination is difficult because of the chemically similar nature of the compounds. However, it is possible to perform this analysis via Near Infrared techniques. When two isomers crystallize together the form site defects which change the nature of the crystal lattice. The lattice in turn affects the hydrogen bonding which can be monitored by diffuse reflectance. The nature and limitations of thisanalysis will be presented.

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SOME APPLICATIONS OF NEAR INFRARED ANALYSIS IN GLASSWOOL INDUSTRY

Dr. R. FUGIER, ISOVER SAINT-GOBAIN, France.

As in most manufactures, glasswool industry has to control its raw materials, intermediate matters and finished goods in order to be sure of the quality of what it sells.

Near infrared spectroscopy was tested to achieve a part of this task.

A great deal of our products is composed of glass fibers bonded by a phenolic resin modified by a lot of additives.

We used Technicon Infraalyzer 400 to titrate some of these additives and to control initial charges of reactors for the synthesis of phenolic resins.

Results show that these works could be done but in any case a standard had to be used before performing measurements.

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IMPLEMENTATION OF NIR AS SCREENING AID FOR QUALITY ASPECTS OF FOOD

P. A. DE LEZENNE COULANDER, FOOD INSPECTION SERVICE, THE NETHERLANDS

The task of the Food Inspection Service in the Hague is, alike the other fifteen regional Food Inspection Services in the Netherlands, to inspect the food production-, trading-, and consumption sites and to sample food for further analysis. All work is based on the Dutch "Warenwet" (Commodity Law). The main objectives are to protect human health and to maintain fair trading. Knowing that only an extremely small fraction of the consumed articles can be sampled, the effectiveness of our work lies in repressive control.

In general the samples analyzed, randomly taken and representative for the whole production, show that it is relatively easy to prepare food obeying the enforced criteria with respect to main constituents. Usually it is a waste of time to analyse those components in every sample with the classical methods as described in the law.

NIR is efficiently used for screening some major components in foods such as fat in minced meat, cheese and some other products, and measuring the water-protein ratio in meat products, the alcohol content of beer, wine and liquors, and the wort extract of beer.

The conversion from the classical methods to NIR procedures has been made as straightforward as possible: namely preparing a calibration curve using the classical methods that suit our needs , preatreating the sample similarly and then applying NIR. The quality of the calibration curve is checked using the samples that show data close to or beyond the boundaries set in the law.

Although the accuracy obtained by NIR is usually not in the same order as that obtained by classical methods, which in our circumstances is probably due to the wide variation in composition of our samples, the use of NIR is very effective with respect to our screening purposes. Only those samples require further analysis with classical methods that provide NIR results approaching or exceeding the set boundaries. Legal action is only taken using the classical methods as described in the law.

Data demonstrating above is presented, including the general information of the calibration curves.

An extensive effort to measure the fat content in milk showed that the long term accuracy of the results obtainable by NIR in our sample population was not good enough to match the producer's capabilities to prepare milk near the set boundaries. For our purposes the desired accuracy for fat in milk by NIR must be 2 % relatively or better, which in our case was not yet obtainable for this type of product.

A much better situation exists in the case of alcohol in beer, wine and liquors. The accuracy obtained is sometimes exceeding the required accuracy for our screening purposes and comes close to the accuracy obtained by distillation and density. Especially this application showed to be a success both with respect to our objectives and to the potentials of NIR.

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THE CONTROL OF COCOA PROCESS

J. PONTILLON, BARRY, FRANCE.

The Barry Group uses InfraAlyzer 400 and will shortly use I/A 350 in their production units in Europe and Africa. An American subsidiary is equipped with an InfraAlyzer 500 for research.

On cocoa crude, cocoa meals and cocoa powders, the systems analyse water content and fat. The mother calibrations were transferred without problems.

On chocolates, milk chocolates and imitations, the same analyses are performed. Limited tests prove that saccharose can be measured. On the other hand, there is little chance to measure lactose ; this may be due to the diversity of milk powders. A good transferability of the calibrations was obtained.

On cocoa butter butter, it is possible to measure free acidity, but this is of little interest. It is possible to measure on liquid the hardness defined as the percentage of solid determined by NMR after a given cycle of crystallization. The calibration coefficients are very high and the calibration cannot be transferred.

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USE OF NIRS FOR THE QUALITY ASSURANCE IN THE PRODUCTION OF SPECIAL TYPES OF FOOD : DIETETIC AND BABY FOODS

MESSRS. F. TACCANI, BANFI, FABBI, M. QUINTIERY, PLASMON (HEINZ), ITALY.

In Italy, dietetic and early childhood products have to report on the label not only the list of ingredients but the exact composition too, with a series of analytical values which have to be constantly respected according to specific tolerances defined by the Ministry of Health for any kind of determination. Ofter, the analytical items indicated on and to be respected are a lot (sometimes more than a decine) and complicated.

The observance of what d'clared requests, for instance, a very onerous job and consequently the preparation of laboratories purposely organized and well equipped.

The research of analytical automatic systems have always been for Plasmon Laboratories one of the main goal and one of the needs more important.

Plasmon Laboratories started with continuous flow automatic systems and, at present, they work with the most advanced and efficient control systems by the NIRS analysis.

It will be described a control system coordinated in the 3 productive factories and the first experiences carried out in as far as controls for some analytical parameters related to childhood cakes, meals, raw materials and milk formulated are concerned.
Some hypothesis on future programs are considered referring to the control of processing and to the control of finished goods suitability.

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PERFORMANCE OF THE INFRAALYZER 400 D ON ICE CREAM

C.D. USHER, UNILEVER RESEARCH, COLWORTH LABORATORY, U.K.

Present manual methods for the analysis of ice cream mix are slow and laborious.

More rapid mid infra-red milk instruments (IRMA) have been used but suffer from two main disadvantages, ice cream must be diluted and separate calibrations are required for different fats.

In a short trial the Infra-Analyzer 400D has been shown to be satisfactory for the direct analysis of ice cream. No dilution is required and a single calibration is sufficient to predict fat in ice cream containing butter fat, palm or coconut oil.

The standard error of prediction (SEP) for four parameters was established on a mixed prediction set of 15 samples and precisions (pooled standard deviations) by the repeated analysis of a standard ice cream mix on five separate occasions.

	SEP	Precision
Protein	0.1 - 0.15	0.026
Total Solids	0.2	0.049
Fat	0.15 - 0.2	0.021
Carbohydrate	0.3 - 0.4	0.083

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SOME APPLICATIONS OF NIRS IN A FOOD RESEARCH INSTITUTE

A.M.C. DAVIES, AFRC, INSTITUTE OF FOOD RESEARCH, NORWHICH LABORATORY, U.K.

Work on the application of NIRS to the rapid analysis of food has been in progress at the Institute of Food Research since 1980. The initial research was carried out on an InfraAlyzer 400 and this lead to the investment in a scanning spectrometer in 1982. The work can be summarised under three headings:- 1) regression analysis applications of NIRS to food constituents, 2) investigation of new mathematical techniques for NIRS and 3) application of NIRS to on-line process control. This talk will concentrate on particular applications based on regression analysis.

The absorption of NIR energy by food constituents is mainly due to C-H, O-H and N-H bonds and the application of NIRS is summarised under these broad headings in Table 1. Emphasis will be placed on the analysis of wine, pea flour, mayonnaise and packaging laminates.

Table 1. Applications of Near Infrared Spectroscopy in Food Analysis

Matrix	C-H	O-H	N-H
General	oil/fat	water	protein
Pea flour	lipid	starch	protein
Mayonnaise	oil		
Salad cream	oil		egg
Coffee	oil	chlorogenic acid	caffeine
Packaging	polythene polypropylene		nylon
Cereals		Non-starch polysaccharides	
Jam/jelly		sugars	
Wine		alcohol	

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EXPERIENCES OF IN-LINE MEASUREMENT AND CONTROL OF BEER ORIGINAL GRAVITY SUMMARY

DR. F.H. WHITE, BASS BREWING RUNCORN LIMITED, U.K.

Substantial cost savings are made with the brewing industry by producing beer at high original gravity (concentrated) and subsequently diluting to sale strength with deaerated water prior to packaging.

To maximise savings and to ensure optimum product quality and consistency, accurate control of the dilution process is essential.

This paper describes the successful application of a Technicon InfraAlyzer 400 to on-line measurement of beer original gravity within a production environment at Bass Runcorn Brewery. Full details of the sample loop and plant modifications are given together with a description of the Technicon flow-through sample cell.

The practical difficulties experienced during the development of the system are discussed and possible future enhancements which would make the sample loop more robust outlined. Finally, the opportunity to utilise the InfraAlyzer result output to give an accurate control of beer dilution is considered.

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Compositional Analysis of minced meat by NIR spectroscopy

mr.A.Frankhuizen, Automation of Analysis, State Instituut for Quality Control of Agricultural Products, Wageningen, the Netherlands

Part of the activities of the State Institute for Quality Control of Agricultural Products consists of developing and promoting automated methods to measure quality parameters in foods and feeding-stuffs. In this framework the suitability of NIR to measure the chemical composition and the product specification of minced meat has been evaluated. This study shows that sample preparation plays an important role in meat analysis. Particle size, homogeneity and temperature of the samples must be standardized.
Using the best combinations of three NIR-filters, useful regression curves were developed to determine moisture, fat and protein in minced meat. Determination of product species, even qualitatively, is hardly possible when using the current NIR equipment.
Details and results of this feasibility study will be presented.

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APPLICATION OF PRINCIPAL COMPONENT ANALYSIS TO THE QUALITY EVALUATION OF GRASS SILAGE BY NIR REFLECTANCE

G. DOWNEY [1], D. BERTRAND [2], P. ROBERT [2] AND M.F. DEVAUX [2]

1. DEPARTMENT OF FOOD SCIENCE AND TECHNOLOGY, AN FORAS TALUNTAIS, KINSEALY RESEARCH CENTRE, DUBLIN, IRELAND.

2. LTAA, INRA, NANTES, FRANCE.

NIR reflectance spectra contain large quantities of information. Much of it is redundant and all spectral data are highly intercorrelated. In addition, a considerable portion of the spectral variation between different samples of any material is due to particle size effects rather than compositional differences. The result of applying multiple linear regression analysis to unmodified spectral data is the generation of a number of calibration equations which may differ markedly in their accuracy and in the wavelengths included. Selection of the best calibration must therefore be made following evaluation of a number of these using a different (prediction) set of samples.

Principal components analysis (PCA) reduces the degree of intercorrelation between n.i.r. spectral data via the creation of synthetic variables ; in this work, PCA was preceded by modification of the raw spectral data so as to minimise particle size effects and restrict the number of wavelengths being investigated. Prediction equations for crude protein (CP) and in vitro digestibility (IVDMD) were then developed by multiple linear regression analysis of the principle components.

Using unmodified spectral data, the best standard error of prediction (SEP) for CP and IVDMD was 0.85 and 3.36 respectively ; applying PC analysis to modified data produced SEP values of 0.65 and 2.72 respectively. Only a single equation for each constituent was developed using the latter approach and assignment of wavelengths included was more satisfactory.

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THE CONTROL OF IN-VIVO DIGESTIBILITY OF FORAGES

A.J.H. VAN ES, J.H. WOLSINK AND H.J. VEDDER

The nutritive value of feeds for livestock depends on composition (organic matter, crude protein, crude fat, crude fibre, starch and sugars) and on the digestibility of these components. The latter property is best measured with digestibility trials with animals (sheep, pigs, poultry) but these last long and require much work. In the last 20 years in-vitro procedures have been developed which simulate animal digestion rather well. These can be used for large series of samples and require much less time. Thus this technique was used to collect the many samples of forages needed for deriving robust NIRS prediction equations.

Data on the precision obtained, both with regard to composition and digestibility of fresh grass, maize silage and wilted grass silage will be presented.

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QUALITY CONTROL OF FEED IN A LARGE COOPERATIVE GROUP

C. BERNARD, UCAAB, FRANCE.

UCAAB, the largest cooperative for the production of animal feed in France, have followed a quality improvement policy for many years.

For this purpose, UCAAB use a large central laboratory and near infrared reflectance systems are used in the main cooperative plants.

The central laboratory does the research and developments on an I/A 500, transfers the calibration to the I/A 400 and I/A 450, provides technical support with personnel training and control of systems with regular ring tests.

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THE USE OF NIRS IN A LARGE FEED COOPERATIVE

ASSOCIATION

P. PETERSEN, L.A.G., DENMARK.

1. L.A.G. : organisation structure, market placement and total turnover.
2. Background for selecting the NIRS method.
3. Background for selecting the Technicon NIR.
4. Controlling of control - and development work -
   How is this made in practice ?
5. At which raw and manufactured goods do L.A.G. make analysis ?
6. The results achieved until now after start in the harvest 1985 ?

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CHOICE OF SPECIFIC WAVELENGTHS FOR GLUCOSINOLATES ANALYSIS IN WHOLE RAPESEED BY NEAR INFRARED REFLECTANCE SPECTROSCOPY.

M. LILA & V. FURSTOSS, STATION D'AMÉLIORATION DES PLANTES FOURRAGÈRES, FRANCE.-

Near Infrared Reflectance Spectroscopy, as a rapid method, is really attractive for quality breeding on many species. After successful calibrations on whole rapeseed grains for oil and protein, we tried another one for glucosinolate content.
This was carried out on five various populations. The optimal wavelengths selected (1674 - 1660 - 1618 - 1650 - 1632 nm) from the best representative population allow sufficient predictions for the other four ones. But it was impossible to use the same equation for glucosinolate analysis on several samples harvested in different years. For three populations we had to do a bias and slope adjustment, and for the last one we have to adjust each equation coefficient. The value of the standard error of the prediction varies from 7.8 to 15.6 µ mole/g while reference analysis error is about 8 µ mole/g. To avoid important error increase due to wavelength variations, we tested each population. Each wavelength was increased and decreased (+/- 4 nm) then forced into calibration calculation. This allow us to note the standard error of estimate variation versus wavelength variation. Specific filters must be now manufactured to use the routine apparatus (with a filter wheel - Technicon 400), so it will be possible to analyse simultaneously proteins, oils and glucosinolates on whole rapeseed.

Additional key word : Brassica napus L., whole rapeseed.

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DEVELOPMENT AND PRACTICAL USE OF NIRS ON GRAINS AND DERIVATES

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The wide range of materials in this field makes it necessary to combine products with less accuracy, instead of making calibrations for each single product. The calibrations thus obtained are more robust and less sensible for changes in the products.

Attention has be taken to the number of filters and height of he factors used in the various calibrations. The influence of the factors and number of used filters concerning the possibility of transfering calibrations between machines has been investigated.

An extract of the results in practical use for the main products on differences with the chemical methods will be given.

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EXPERIENCE OF NIR AS METHOD FOR PROTEIN PAYMENT IN WHEAT TO SWEDISH FARMERS

H. JOHANSSON, SWEDISH CEREAL LABORATORY, SWEDEN.

Swedish farmers have since many years been paid a premium for protein content in wheat. About one third of them, mainly those in southernmost Sweden, have been paid premium for the protein content in each individual delivery. Farmers in middle Sweden have accepted protein premium according to the mean value in the region or in their farmers cooperative although each farmer could request for individual protein premium. The protein premium during the two last years has been three per cent for winter wheat and four and a half per cent for spring wheat per percentage point protein above eleven per cent. The Kjeldahl method has been used with very few exceptions up to 1984.

The Swedish Association of Cereal Chemists established late in 1982 a committee for standardization of the NIR technique as a method for protein determination on farmers deliveries. In the summer 1984 the Swedish Agricultural Marketing Board authorized the Swedish Grain Trade Association to accept NIR for protein determination in wheat during the crop season 1984/1985 if the instructions given by the NIR committee of the Swedish Association of Cereal Chemists were followed. Almost the same instructions were given for the crop season 1985/1986. More than 100 000 deliveries of wheat from the harvest 1985 have been analyzed with about fifty NIR instruments. Experience from the introduction of the technique and the results from the monitoring system will be discussed.

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QUALITY CONTROL OF CEREALS IN SPAIN

DR. C. RIVAS, SENPA, SPAIN.

The National Service for Agricultural Products (SENPA) is the intervention body for cereals.

In this lecture, we will tell about the evolution of intervention in Spain and the quality control introduced until the entry of Spain in the E. E. C.

We had to install 27 laboratories and use the N I R technique to measure moisture and protein percentages.

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APPLICATION OF MULTIDIMENSIONAL ANALYSES IN N.I.R. SPECTROSCOPY

BERTRAND D., ROBERT P., DEVAUX M.F., GRAPPIN R.
I.N.R.A., FRANCE.

The main objective of chemical laboratory analyses is to help the practician to take a correct decision about the best industrial utilization of studied products. The usual application of NIR spectroscopy involves two steps 1) Prediction of chemical values 2) Examination of these estimated values to direct the product towards a particular application. The transformation of NIR spectral data into chemical values may be a complex process, particularly when the reference laboratory methods are time consuming or not very relevant. The present communication describes different attempts to reduce the role of the reference manual methods in NIR spectroscopy through a direct examination of spectral data. Statistical multidimensional analyses allow to give comprehensive description of sets of numerical values such as NIR spectral or chemical data. Two kind of multidimensional analyses are described : Principal Component Analysis (PCA) and Correspondence Analysis (CA). PCA and CA work on rectangular tables, the rows of which are called "observations" and the columns "variables". These analysis are able to give graphical representation of data which are easy to understand. PCA produce two kind of graphs : correlation circles, which are representation of intercorrelations between variables, and factorial representations of observations. Examples of correlation circles representing NIR spectral data, are given. When chemical data are available, it may be useful to place them as supplementary variables; this is a simple and rapid way to see if they can be predicted by NIR data. Factorial representations of observations provide a way to estimate the similarity between several observations. In some case, the spectral similarities correspond to biochemical or qualitative likeness. Examples of such cases is given (milk products, forages).

CA produce graphs where variables (wavelengths) and observations (spectra) are represented together. Neighbouring of variables and observations can be generally interpreted. For examples, in an experiment on milk products, wavelengths which were caracteristic of fat were neighbouring with products containing a large amount of lipids.

The possibilities of use of NIR spectroscopy with only limited references to manual analyses are discussed.

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A COMPARISON OF NIRA AND CLASSICAL IR ANALYSIS, Tomas Hirschfeld
Lawrence Livermore National Laboratory, Livermore, California

Near infrared reflectance analysis, using a combination of diffuse reflectance and correlation spectroscopy is fast encroaching upon the quantitative application of the more common mid-infrared spectroscopy. While clearly unsuitable for qualitative analysis because of high spectral densities and scarce spectral reference data, NIRA gives precise analysis of quite difficult to measure or to intepret samples. Methods development for NIRA requires a large collection of preanalyzed samples, but tends to be less dependent on spectral properties or even on a well defined chemical question. Once the rather difficult transition between working methods has been made, NIRA should often be the technique of choice for quantitative analysis. An analysis of the weaknesses of each method is used to establish the domain of each.

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THE FUTURE OF NIRS IN AGRICULTURE

DR. IR. R. BISTON

The evolution of agricultural production (E.E.C. quotas) and the development of international exchanges dictates to the official bodies a control more and more strict of the quality.

Thus, NIRS has and will have a place more and more preponderant

Its rational application needs to standardize the method and to research universal calibrations accessible to all users.

In the field of agronomical research, it allows to research ways impossible with other methods.

The analytical potential of this technology is necessary every day thanks

- to a better specification of the systems ;
- to the development of instrumentation more suitable ;
- to software more and more powerful.

One can predict for the near future analytical spectrometry coupled to a data bank which will reduce the tedious calibration work.

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THE FUTURE OF NIRA: WHERE IS IT GOING? T. Hirschfeld, Lawrence Livermore Laboratory Livermore, California 94550.

The tremendous popularity of NIRA in agricultural and food chemistry is not yet matched by a comparable usage among industrial analytical spectroscopists. As realization of the intrinsic advantages of the near IR region for quantitative analysis becomes widespread, this area will quickly dominate the overall field of NIRA.

In this more general application, competitive ease of use of NIR in comparison with the classical mid-IR spectroscopy will be required. This is particularly so in view of the sophistication (and power) of the learning algorithm used in NIRA. The simultaneous increase in our understanding of the mathematics of NIRA and the power of microcomputers is enabling us to put more and more of the skills involved in the technique into the program. This gradualist approach to artificial intelligence methods in analytical chemistry already enjoys wide practical application. NIRA has already achieved the benchmark analysis speed of 20 seconds from sample bottle to result printout. In the future, full automatic method development is an exciting further near term potential.